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COMPLETE SPECIFICATION

A Method of and Apparatus for Removing Monomeric Constituents from Polymerised Lactams

We, INVENTA A. G. FÜR FORSCHUNG UND PATENTVERWERTUNG, a corporate body organised under the laws of Switzerland, of Kapellplatz 9, Lucerne, Switzerland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

As is well known the polymerisation of monomeric lactams having more than 6 members in the ring does not proceed to completion but ends in a state of equilibrium with a greater or less proportion of the monomeric starting material. Thus, for example, if the starting material used is ε-aminocaprolactam there is obtained in the final stage of the polymerisation a mixture of about 90% of high polymeric material with about 10% of monomeric, dimeric, etc., lactams. It has not hitherto been possible appreciably to displace the state of equilibrium towards the high polymer side, either by altering the conditions under which the polymerisation is effected or by the addition of accelerators of any kind. Since too high a proportion of monomeric constituents has a disadvantageous effect not only on the spinning process but also reduces the strength of the finished polyamide silk, fibres or bristles, the removal of the monomers is necessary. For this purpose two methods have previously been used, namely vacuum extraction of the molten polyamide after polymerisation has been completed or extraction of the polyamide pieces or chips with water (see Swiss Patent No. 230,271). With the latter method it is possible to lower the content of monomeric constituents to about 1%, which is hardly possible by vacuum extraction, because if the melt remains for some time in the liquid state the monomers are re-formed. Also, if chips containing 1% of monomeric constituents are re-melted, as is necessary for spinning, a re-formation of monomers occurs to an extent which is dependent on the period during which the melt remains in the spinning head. If the delivery of the feed pump is very small, as is the case when spinning threads of fine titre, the re-formation of monomers may increase up to about 7%. When threads of coarse titre are spun and the pump delivery is correspondingly greater, the re-formation of monomers may be up to 3.5% to 4%. Thus, neither by the difficult process of extraction with water nor by vacuum extraction is it possible to obtain a polyamide melt which is sufficiently free from monomers immediately before it reaches the spinning nozzle. The removal of the monomers to an extent depending on the kind of fibres to be spun is desirable because it not only facilitates the spinning process, but also produces a spun material in which the content of monomers is a minimum. Until now the removal of the monomers from the melt was very unfavourable from an economical point of view. Thus, for example, the high polymers of ε-aminocaprolactam which have hitherto been used in large quantities contain about 9% of monomers, which must be removed by extraction, and from which about 90% can be recovered by evaporation of the water used for the extraction. On melting the polyamide material in the spinning apparatus, there is re-formed on the average about 5% of low polymer material which is reduced to not more than 2% by washing the spun polyamide silk whereby 3% is lost since the working-up of the washing water is not economical. About ¾% is lost as vapour so that the effective loss amounts to approximately 4%.

The present invention enables the loss

of monomeric constituents to be largely avoided. The polyamide melt and the threads or bristles produced from it also have a considerably lower content of monomeric consituents than previously when the process and apparatus of the invention are used.

According to the invention, the polymerised material is converted into the form of a number of threads which are maintained in a liquid state, and from which the monomeric material is allowed or caused to evaporate under normal or reduced pressure.

Apparatus for carrying the process of the invention into effect comprises a chamber provided with heating means having near one end a nozzle provided with a number of orifices and a pump by which the molten polymerised material may be pumped through the nozzle and near the other end a sump into which the threads of liquid from the nozzle fall by gravity, the chamber having an outlet for the vaporised monomers and preferably an inlet through which an inert gas may be introduced.

In order that the invention may be easily understood and readily carried into effect, an apparatus for carrying out the process of the invention is illustrated diagrammatically and by way of example in the accompanying drawing, which shows a section through the apparatus.

Referring to the drawing the polyamide melt, of e-aminocaprolactam polymerisate which can be supplied directly from the reaction vessel or in the form of a mixture from several reaction vessels, is introduced into the receiver 1. It is, of course, also possible to start with solid material, for example chips, which are melted and then delivered to the receiver 1; extraction of the monomers from the solid material before melting is not necessary. The material is squirted by means of the pump 2 through an auxiliary nozzle 4. The nozzle 4 is a kind of spinneret having a number of fine holes, the number of which depends on the delivery pump which depends on the titre of the silk. The object of this auxiliary nozzle is to give the polyamide melt which is forced through it as large a surface as possible in the form of threads which are still in a molten condition, in order that the superfluous monomers may evaporate while falling through the tube 5. This evaporation can be accelerated by introducing oxygen-free nitrogen, preferably in small quantity, through the tube 12. The monomers which distil off are collected in the chamber 11 into which they pass through the pipe 10. The tube 5 has a double wall (which is not illustrated) and it is maintained at a temperature such that on the one hand the monomers which distil off are not condensed, and on the other hand the threads which collect in the vessel or sump 6 are still in the molten state. The receiver 1, the chamber 3 and the pump 2 are also heated by a heating jacket (which is also not illustrated) the purpose of which is to supply heat to the melt in accordance with the amount of heat withdrawn by the heat of evaporation of the monomers. The sump 6 is so small that the melt remains in it only until it is sufficiently degasified, whereby the reformation of monomers is limited to a minimum. The depth of the material in the sump 6 can be automatically regulated by means of one or more contacts 9 arranged at various heights. These contacts are electrical contacts and they extend into the walls of the vertical chamber. The regulation of the depth of the material in the sump 6 is preferably effected by a conductivity measuring device which is based on the fact that the conductivity of the melt in which the contact is immersed when the depth of the material in the sump is considerable, is greater than the conductivity of the air. By means of the contact an impulse is transmitted to the drive of the pump 2, which is accordingly driven more slowly or more quickly, so as to alter the amount of its delivery. The material from the sump 6 is spun in the usual manner being delivered by the pump 7 to the spinneret 8. It has been found that the spinning process described proceeds more smoothly than was previously the case, and the tendency of the threads emerging from the spinneret to crinkle is considerably less.

What we claim is:—

1. A process for removing monomeric consituents from polymerised lactams before spinning, according to which the polymerised material is converted into the form of a number of threads which are maintained in a liquid state, and from which the monomeric material is allowed or caused to evaporate under normal or reduced pressure.

2. A process according to claim 1, in which the polymerised melt is conducted directly from the polymerisation vessel to the apparatus in which it is converted into threads.

3. A process according to claim 1, in which solid polymerised material is supplied in the form of chips or pieces to the apparatus in which it is converted into threads.

4. A process according to any preceding claim, in which evaporation of the

monomeric constituents is promoted by passing an inert gas over the threads.

5. A process according to claim 4, in which oxygen-free nitrogen is used as the inert gas.

6. A process of spinning polymerised lactams which comprises converting the polymerised material into the form of a number of fine threads which are maintained in a liquid state, evaporating monomeric materials from the threads, collecting the liquid from which monomeric material has been evaporated in a sump, and delivering the material from the sump to a spinneret.

7. An apparatus when used for carrying out the process claimed in any preceding claim, comprising a chamber provided with heating means having near one end a nozzle provided with a number of orifices and a pump by which the molten polymerised material may be pumped through the nozzle and near the other end a sump into which the threads of liquid from the nozzle fall by gravity, the chamber being provided with an outlet for the vapourised monomers and preferably an inlet through which an inert gas may be introduced.

8. An apparatus in accordance with claim 7, in which the chamber is in the form of a tube.

9. An apparatus according to claim 7 or 8, in which the chamber is provided with a double wall.

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1 SHEET

This drawing is a reproduction of the Original on a reduced scale.